

that has been shown to work. Where alkaline particulate matter and condensed moisture are present in the gas stream, the filter shall be heated above the moisture dew point but below 225 °C (437 °F).

16.3.2 Sample Recovery. Recover the sample according to Section 8.4 except for discarding the contents of the midget bubbler. Add the bubbler contents, including the rinsings of the bubbler with water, to a separate polyethylene bottle from the rest of the sample. Under normal testing conditions where sulfur trioxide will not be present significantly, the tester may opt to delete the midget bubbler from the sampling train. If an approximation of the sulfur trioxide concentration is desired, transfer the contents of the midget bubbler to a separate polyethylene bottle.

16.3.3 Sample Analysis. Follow the procedures in Sections 11.1 and 11.2, except add 0.5 ml of 0.1 N HCl to the Erlenmeyer flask and mix before adding the indicator. The following analysis procedure may be used for an approximation of the sulfur trioxide concentration. The accuracy of the calculated concentration will depend upon the ammonia to SO₂ ratio and the level of oxygen present in the gas stream. A fraction of the SO₂ will be counted as sulfur trioxide as the ammonia to SO₂ ratio and the sample oxygen content increases. Generally, when this ratio is 1 or less and the oxygen content is in the range of 5 percent, less than 10 percent of the SO₂ will be counted as sulfur trioxide. Analyze the peroxide and isopropanol sample portions separately. Analyze the peroxide portion as described above. Sulfur trioxide is determined by difference using sequential titration of the isopropanol portion of the sample. Transfer the contents of the isopropanol storage container to a 100-ml volumetric flask, and dilute to exactly 100 ml with water. Pipette a 20-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 0.5 ml of 0.1 N HCl, 80 ml of 100 percent isopropanol, and two to four drops of thorin indicator. Titrate to a pink endpoint using 0.0100 N barium perchlorate. Repeat and average the titration volumes that agree within 1 percent or 0.2 ml, whichever is larger. Use this volume in Equation 6-2 to determine the sulfur trioxide concentration. From the flask containing the remainder of the isopropanol sample, determine the fraction of SO₂ collected in the bubbler by pipetting 20-ml aliquots into 250-ml Erlenmeyer flasks. Add 5 ml of 3 percent H₂O₂, 100 ml of 100 percent isopropanol, and two to four drips of thorin indicator, and titrate as before. From this titration volume, subtract the titrant volume determined for sulfur trioxide, and add the titrant volume determined for the peroxide portion. This final volume con-

stitutes V_i, the volume of barium perchlorate used for the SO₂ sample.

17.0 References

1. Atmospheric Emissions from Sulfuric Acid Manufacturing Processes. U.S. DHEW, PHS, Division of Air Pollution. Public Health Service Publication No. 999-AP-13. Cincinnati, OH. 1965.
2. Corbett, P.F. The Determination of SO₂ and SO₃ in Flue Gases. *Journal of the Institute of Fuel*. 24:237-243. 1961.
3. Matty, R.E., and E.K. Diehl. Measuring Flue-Gas SO₂ and SO₃. *Power*. 101:94-97. November 1957.
4. Patton, W.F., and J.A. Brink, Jr. New Equipment and Techniques for Sampling Chemical Process Gases. *J. Air Pollution Control Association*. 13:162. 1963.
5. Rom, J.J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Office of Air Programs, U.S. Environmental Protection Agency. Research Triangle Park, NC. APTD-0576. March 1972.
6. Hamil, H.F., and D.E. Camann. Collaborative Study of Method for the Determination of Sulfur Dioxide Emissions from Stationary Sources (Fossil-Fuel Fired Steam Generators). U.S. Environmental Protection Agency, Research Triangle Park, NC. EPA-650/4-74-024. December 1973.
7. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, PA. 1974. pp. 40-42.
8. Knoll, J.E., and M.R. Midgett. The Application of EPA Method 6 to High Sulfur Dioxide Concentrations. U.S. Environmental Protection Agency. Research Triangle Park, NC. EPA-600/4-76-038. July 1976.
9. Westlin, P.R., and R.T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards. *Source Evaluation Society Newsletter*. 3(1):17-30. February 1978.
10. Yu, K.K. Evaluation of Moisture Effect on Dry Gas Meter Calibration. *Source Evaluation Society Newsletter*. 5(1):24-28. February 1980.
11. Lodge, J.P., Jr., *et al.* The Use of Hypodermic Needles as Critical Orifices in Air Sampling. *J. Air Pollution Control Association*. 16:197-200. 1966.
12. Shigehara, R.T., and C.B. Sorrell. Using Critical Orifices as Method 5 Calibration Standards. *Source Evaluation Society Newsletter*. 10:4-15. August 1985.
13. Curtis, F., Analysis of Method 6 Samples in the Presence of Ammonia. *Source Evaluation Society Newsletter*. 13(1):9-15 February 1988.